

SOME TIPS ON TRITIUM TECHNOLOGY

J. L. Anderson, D. O. Coffin, J. E. Nasise, and R. H. Sherman
Materials Chemistry Group, Materials Science and Technology Division

R. A. Jalbert
Radiation Protection Group, Health, Safety and Environment Division

Los Alamos National Laboratory
Los Alamos, New Mexico 87545
(505) 667-1410

ABSTRACT

This collection of practical suggestions should prove useful to workers at tritium installations where gloveboxes and cleanup systems are used, or where upgrades to such systems are anticipated. These tips are effective approaches to meeting today's stringent containment requirements, and include many applications and extensions of accepted tritium practice, plus a few innovations of our own. The subjects covered are tritium piping systems, glovebox operations, personnel protection, tritium monitoring, and contamination control. Some specific problems arising out of recent experience are also discussed, along with some tentative solutions.

INTRODUCTION

We operate two modern tritium facilities at Los Alamos: the Tritium Systems Test Assembly (TSTA), and the Tritium Salt Facility (TSF). Both were designed to meet stringent environmental and personnel protection standards. Besides our recent experience in state-of-the-art plants, we have also worked for many years in older, less rigorously controlled tritium facilities. Although our approaches to meeting today's containment regulations are mostly applications and extensions of accepted tritium practice, we believe that our successful experience handling and containing large quantities of tritium in various forms qualifies us to comment on the application of this technology.

PROCESS PIPING

For primary containment we chose only all-metal components, so that no oils, elastomers, organics or fluorocarbons are exposed to tritium. Primary piping in the TSTA is annealed copper tubing with brazed-on tube fittings of the zero-clearance, coined-gasket type¹. In the TSF the same fittings are used, but here they are welded to 316-L stainless steel tubing. Valves, transducers, and most other components were selected from

all-metal designs commercially available. We consider these to be limited-life components, so we installed them with our standard fittings. This construction differs from the all-welded systems found at nuclear plants, but it enables replacement of a faulty valve or transducer without cutting, welding, or brazing tritium-contaminated tubing. We have demonstrated that a tritium system containing hundreds of demountable components can have a leak integrity rivalling that of an all-welded system. All valves are metal-bellows sealed and pneumatically actuated². The only exception to our no-organics rule is that the valve stems of these air-operated valves are tipped with polyimide.

All tritium-handling pumps are dry, hermetic, and packless. Our basic gas transfer pump is the domestically manufactured metal-bellows compressor³, which we have modified to eliminate all fluorocarbons from the pump chamber. Where high vacuum performance is required, the metal-bellows pump is supplemented by another bellows-sealed vacuum pump with an orbiting scroll geometry (Fig. 1), available commercially from France⁴. Tests of these pumps were reported previously⁵, and we now have considerable tritium experience with them. Both pumps have given flawless, leak-free service with concentrated tritium, both at Los Alamos and elsewhere⁶. The overwhelming advantage of these devices is that they do not contaminate the tritium gas with oil or mercury vapor or with the gaseous decomposition products that result when tritium interacts with organic materials. Since they are relatively small and have no routine servicing requirements, we chose to mount them inside gloveboxes for secondary containment.

GENERAL GLOVEBOX OPERATIONS

Glovebox atmospheres must be inert to the form of tritium being handled, primarily to avoid flammable mixtures in the event of large tritium releases. At TSTA, where tritium is handled only in its elemental gaseous form, we use dry nitrogen in static gloveboxes, with

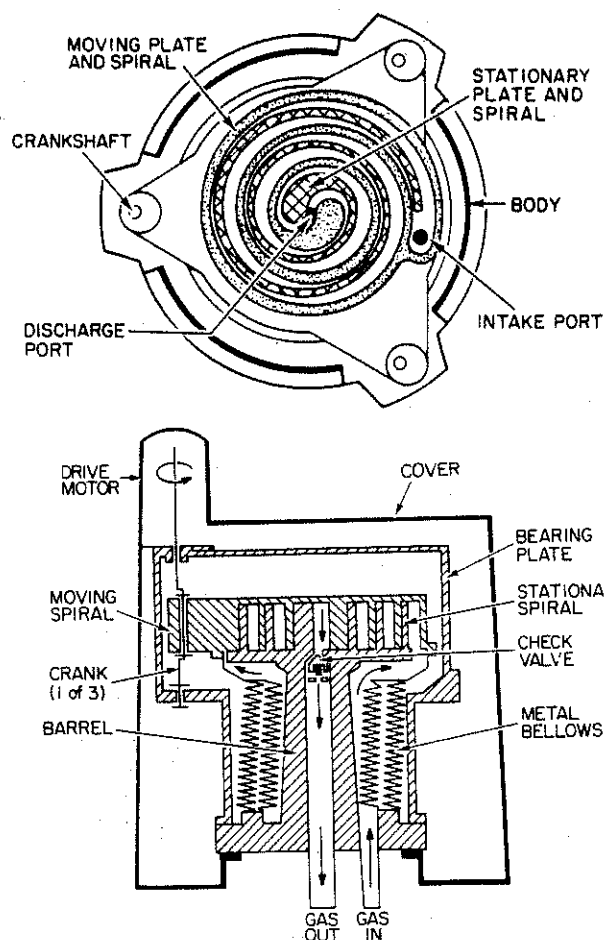


Fig. 1. Tritium-compatible orbiting-spiral high vacuum pump

once-through purging available whenever rising tritium or oxygen concentrations require it (Fig. 2). In the TSF gloveboxes an ultradry inert atmosphere with good heat transfer is required, so helium is continuously recirculated through a chemical dry train with all regenerated gas streams routed to a tritium removal system (Fig. 3). Helium also provides a capability for leak testing of the gloveboxes and of components fabricated therein. All purge and waste gases from the gloveboxes pass through general purpose tritium removal systems before they are vented to the atmosphere.

Instrumentation feedthroughs for gloveboxes are of the multiconductor hermetic type, and fluid feedthroughs are all vacuum grade; no tapered pipe threads are allowed in newer construction. Our preferred feed-through is the International Standards Organization ISO-KF vacuum fitting⁷. These are available commercially in weldable grades of stainless steel and aluminum. We weld several of these flanged stubs into each glovebox before original installation (Fig. 4.A); then feedthroughs of all types--pneumatic, hydraulic, high voltage, high current, instrumentation--are purchased from vacuum component suppliers and clamped to the mating flange already on the glovebox. We modify this same fitting to provide a secondary conduit around tritium piping that must pass between gloveboxes (Fig. 4.B).

TSTA gloveboxes are monitored for tritium with internally mounted ion chambers. For comparison of the internal ion chamber with an external reference each glovebox is equipped

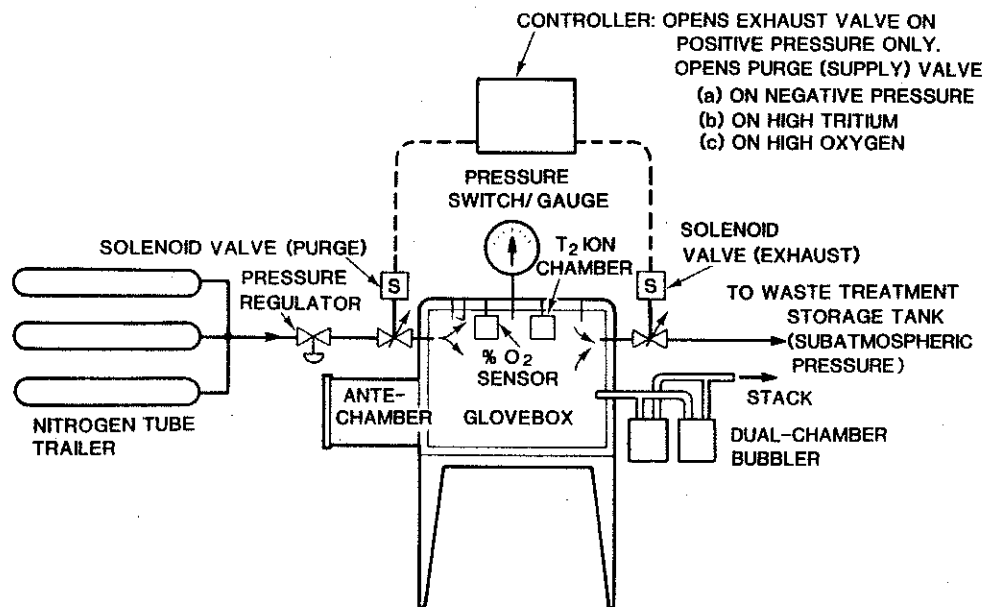


Fig. 2. Once-through nitrogen purging for the Tritium Systems Test Assembly (TSTA).

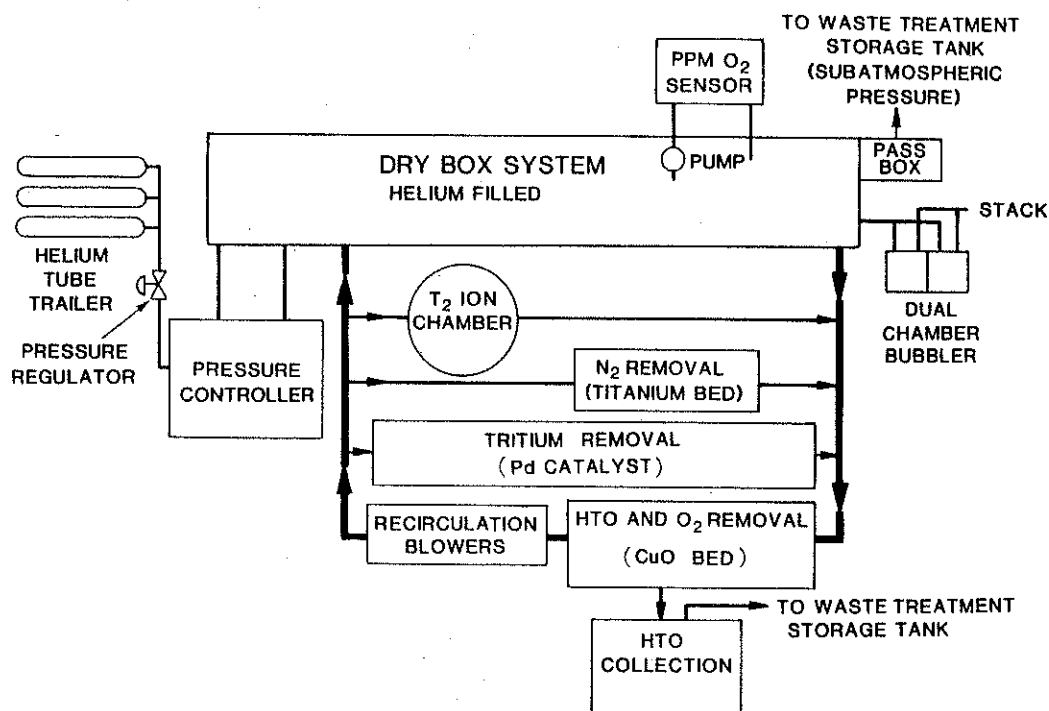
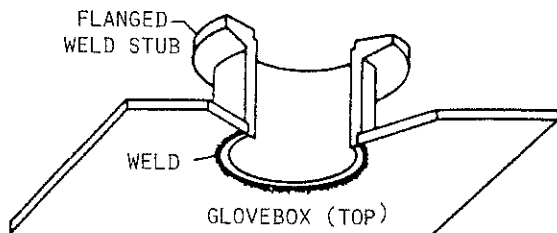
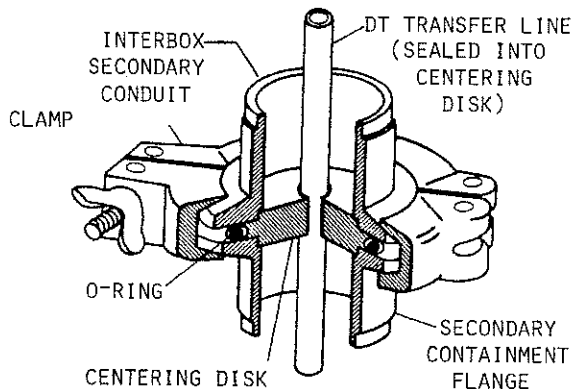


Fig. 3. Full Dry-train recirculation with tritium removal for the Tritium Salt Facility (TSF).



A. WELDED INSTALLATION IN GLOVEBOX



B. ADAPTATION TO SECONDARILY CONTAINED PIPING

Fig. 4. Glovebox feedthrough utilizing a modified ISO-KF quick vacuum flange.

with a pair of quick-disconnect fittings. We have increased the utility of these connections by attaching an internal hose that can reach any point inside the glovebox. When we connect an external pump and tritium monitor we have an ultrasensitive leak detector (Fig. 5). With only 1-2% tritium in the piping we can find leaks as small as 10^{-12} cm³-s⁻¹. Leaks that are orders of magnitude larger than this, but which can barely be detected with a helium leak detector in the sniffer mode, yield tritium detector readings 10 to 100 times the glovebox background and are easily located.

PERSONNEL PROTECTION

At the TSTA all tritium piping is secondarily contained, and the main experimental rooms are maintained at contamination levels suitable for the general public. No protective clothing is required in the work space for routine work on uncontaminated equipment. TSTA is remotely operated, so gloveboxes are entered only for repair operations. For any work in gloveboxes personnel wear lab coats and PVC gloves to reduce contact absorption of tritiated water. Self-contained breathing air packs and two-piece supplied-air suits are available for emergency use in case of a tritium release into the experimental room.

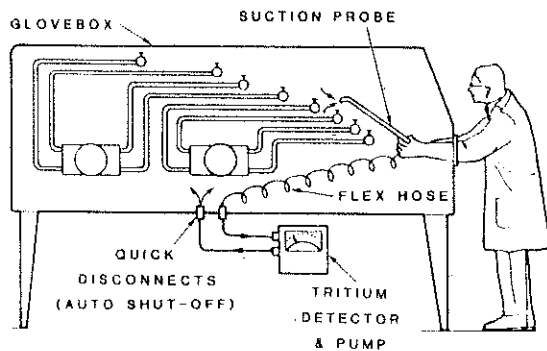


Fig. 5. Leak detecting in a glovebox with an external pump and tritium monitor.

At the TSTF, reactive metal tritides are handled in a large dry box system connected to a dry-train for maintaining an inert atmosphere. Three types of respiratory equipment are available: two-piece supplied air suits; self-contained breathing-air packs; and full-view supplied-air face masks. The suits are used for planned entries into highly tritium contaminated areas. Air packs and facemasks are used in low level tritium contaminated atmospheres in conjunction with lab coveralls, PVC gloves, and booties. When immediate entry is necessary to prevent further contamination or to perform a rescue, the self-contained air packs can be used in emergency.

Breathing air from a tube trailer is supplied through a building manifold to both facilities, and pressure is reduced to 100 psig at each of the breathing air stations. Each station supplies air through 50-ft hoses either to 2 suits at 8 scfm each, or to 2 face masks at 4 scfm each. The station contains an adjustable pressure regulator and pressure gauge, precalibrated to deliver the flow rates above. Alarms ring in both facilities if the air pressure in the building supply manifold drops, indicating imminent exhaustion of the breathing air supply.

MONITORING

We monitor tritium principally by conventional ion chambers. At the TSTA we have eliminated most of the air pumps by placing ion chambers with perforated walls (Fig. 6) in the enclosure to be monitored. We use this technique for all glovebox and exhaust duct monitors, as well as for the high-range room monitors. The high noise levels and all-too-frequent failures associated with mechanical pumps are thus eliminated. Another advantage is that ion chamber contamination is reduced when monitoring widely changing concentrations of tritiated water vapor.

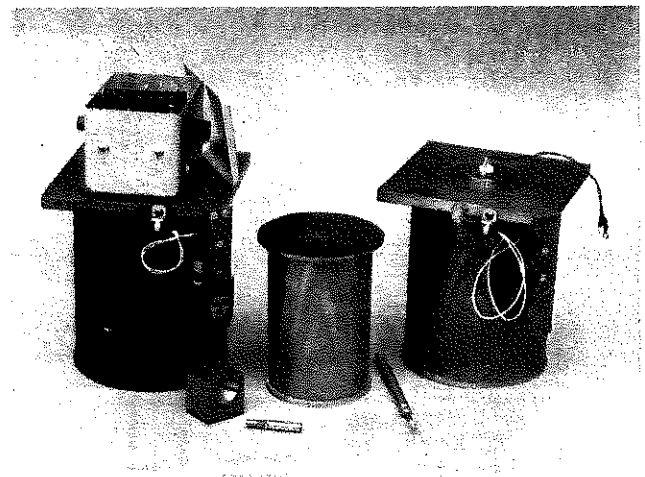


Fig. 6. Open-wall ion chambers with gamma check sources.

Since the TSTA is computer-controlled we use the computer to monitor the air flow of the flow-through ion chambers and the power supplies of most of the tritium ion-chamber instruments. The computer also periodically exposes a gamma check source (Fig. 6) to each low-range monitor to check its response.

We monitor the TSTA stack and exhaust ducts with conventional ion chamber instruments with integrating capability. For integrative passive monitoring of the stack we use a version of the Mound Facility glycol bubbler⁸ to distinguish between HTO and HT.

CONTAMINATION CONTROL

Most gloveboxes are equipped with evacuable airlocked passboxes for facilitating repair and replacement operations. Tritium contaminated process components requiring repair

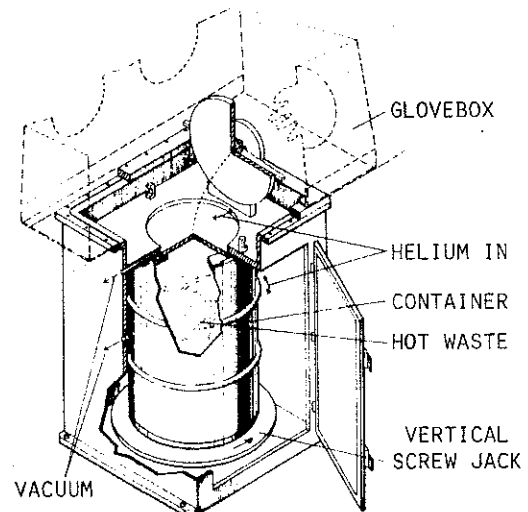


Fig. 7. Solid waste disposal from a glovebox using an airlock in the floor.

or disposal are removed from the system and transferred through passboxes to a glovebox or fume hood dedicated to that function. This repair/disposal glovebox connects to an air-locked hatch that accesses a waste disposal drum (Fig 7). This waste disposal system has been in use at the TSF⁹ for several years and is also installed in TSTA.

We have developed techniques for adding major equipment and enclosures to existing contaminated enclosures without exposing the inside of the contaminated glovebox to the environment (Fig. 8). After removing any floor support or external structure, we seal-weld a ring flange to the unbroken outside face of the glovebox. The flange carries a gasket groove and a pumpout port. Next we bolt on the gasketed glovebox extension containing the add-on equipment, and use the special flange port to purge and backfill the extension with the same gas as in the original box. Finally, an opening is sawed through the center of the flange from inside the original glovebox, using the oxide trace from original welding as a cutting guide. This technique has been particularly useful at the TSF, because the reactive tritium compounds handled there have caused severe contamination of interior surfaces.

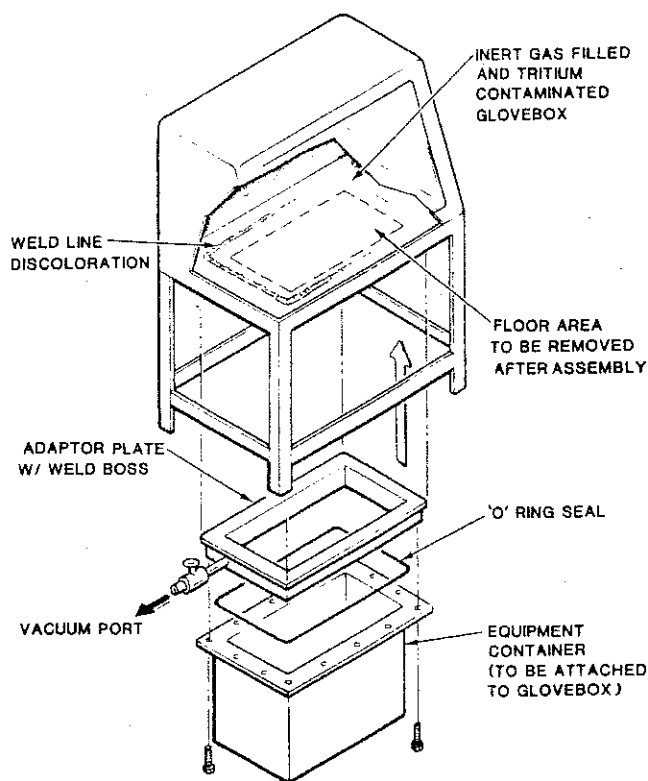


Fig. 8. Method for adding on to a contaminated glovebox enclosure.

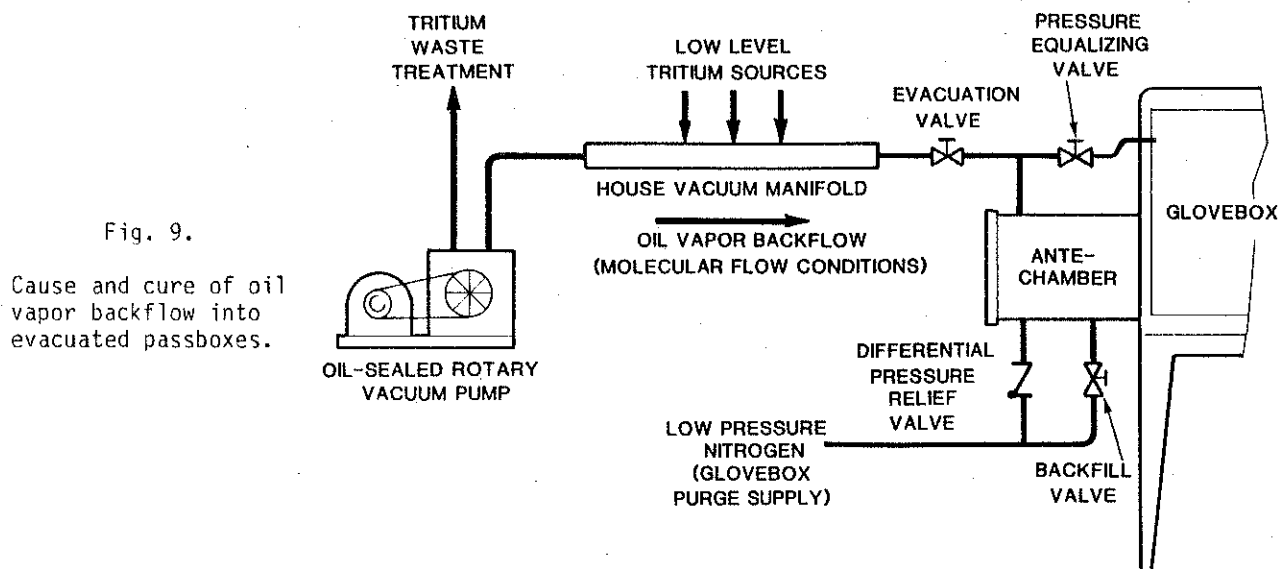
In the TSTA the nitrogen glovebox atmosphere prevents formation of a flammable mixture following a release of process gas, but the oxygen content is still too high (1-2%) to inhibit spontaneous formation of HTO. We have observed that during a steady release of tritium, the ratio of tritium in the oxide form to total tritium may be as high as 1/3. This is an equilibrium ratio, measured while purging with dry nitrogen. If a small quantity of normally humidified room air is admitted to the box, the tritium concentration increases sharply, especially that of the oxide component. This is not surprising, since the H₂O/HTO surface exchange process is far more effective at reducing HTO surface contamination than is simple outgassing into a dry atmosphere.

We have used this principle to advantage for decontaminating passbox interiors without having to open them. We evacuate the passbox and aspirate a little water into it during air backfill. We leave the surfaces exposed to saturated air for several hours and evacuate the passbox again. This process can be repeated until the chamber contamination level is reduced to a level where it can be opened up. After the cleanup, we leave a small quantity of desiccant in the passbox before closing it up again. For oil contamination volatile organic solvents can be aspirated into the enclosure instead of water.

PROBLEMS

Some problems we have encountered are: (a) tritium contamination of our vacuum passboxes by oil vapor backstreaming from the house vacuum system; (b) contamination of gloveboxes by ingestion of oil from bubblers; (c) chronic maintenance difficulties associated with tritium cleanup systems; and (d) some gas chromatography problems in tritium cleanup processes.

The passbox contamination problem is generic to any tritium facility that uses a conventional oil-sealed vacuum pump for the house vacuum system. If this system is used to pump tritium-contaminated lines and components, the pump oil becomes irreversibly tritium contaminated, and under molecular flow conditions this tritium-contaminated oil vapor will back diffuse into any component connected to the pump. The manual evacuation valves on some of our TSTA passboxes were inadvertently left open to the house vacuum manifold during our first month of operation, and severe contamination of the passbox interiors resulted. The best but most expensive cure for this is to use an oil free pump for the house vacuum system. Such vacuum pumps of the moving scroll design are now available⁴. We plan



to eventually separate our house vacuum functions into two systems. A new system served by a small oil-free scroll pump will be used for tritium contaminated evacuation jobs, and the present house vacuum system will be restricted to passbox evacuation and general purpose (uncontaminated) house vacuum services.

Our interim solution to the passbox contamination problem is to ensure that molecular flow conditions are never reached in the vacuum manifold while the passbox is connected to the pump, regardless of how long the evacuation valve is held open. We accomplish this by installing in each passbox a nitrogen leak that opens up whenever the passbox pressure becomes subatmospheric. The leak is not large enough to prevent good dilution by normal purge-evacuate-backfill operations, but is sufficient to maintain viscous flow towards the vacuum pump while the passbox evacuation valve is open (Fig. 9).

The second problem we encountered with gloveboxes early in TSTA operations was that of violent ingestion of oil from pressure relief bubblers. The most common relief bubbler design is a simple U-tube. For changes of glovebox pressure this device relieves the overpressure, not by releasing bubbles as the name implies, but by violently discharging the entire charge of oil from the U-tube. For negative glovebox pressures this results in dispersion of the oil over the interior surface of the glovebox. This oil subsequently absorbs tritium from the glovebox atmosphere and is a chronic source of contamination thereafter. Mound Facility recognized this problem many years ago and designed a double-chamber bubbler to prevent this type of accident. We had used bubblers of the Mound design at our TSF since it went into

operation, and we have now modified this design for our gloveboxes at TSTA (Fig. 10). At the TSTA we have also replaced the bubbler oil with ethylene glycol, which is denser, soluble in water, and advantageous over oil as a bubbler medium in several respects.

We have noted several problems during tritium cleanup operations that are common to all commercially built tritium cleanup systems. Much of the cost of the dryers and catalytic reactors used in cleanup systems is in their high-integrity containers, heaters, and other integral components; yet there is no way to replace the active material when it becomes ineffective, so the entire unit must be thrown away. This equipment should always have provisions for changing the active material. We must now replace an expensive catalytic recombiner in the TSF cleanup system because of poisoned catalyst. Specifications for the new unit do require catalyst change-out capability.

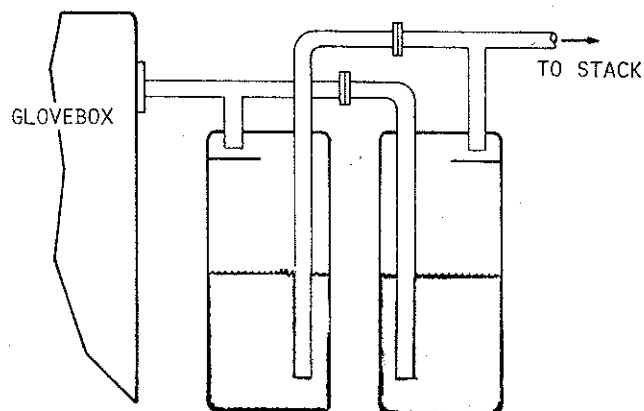


Fig. 10. Anti-suckback pressure-relief bubbler for gloveboxes.

Another generic problem with cleanup systems relates to general serviceability and replaceability of valves, heaters, and other accessory components. We recently experienced total failure of a valve on one of our tower dryers, when its shaft sheared off inside the packing. The valve had been hard-soldered into position in a section of rigid piping, so we had to cut and braze contaminated piping to make a replacement. The problem was exacerbated by the fact that the valve position sensor was attached to the valve actuator rather than the valve itself, so the valve was inoperative for many months before the failure was detected. The lessons here are that critical remote-operated valves should have positive position indication, and they should be capable of being replaced with demountable, zero-lateral-clearance flanges. We currently follow these rules for our tritium process piping, and they should apply to cleanup systems as well.

We chose gas chromatography (GC) as our principle analytical tool in the TSTA. Among the advantages of GC over a mass spectrometer are lower cost, simplicity, reliability, ease of operation and maintenance, and ready interfaceability to computers. One unanticipated problem grew out of an interaction between chromatographs used concurrently for different applications. A typical GC analysis in the TSTA Tritium Waste Treatment (TWT) system uses a molecular sieve column and helium carrier gas to separate total hydrogens from nitrogen, oxygen, etc. At the same time some of our process systems are using GCs with cryogenic columns and neon carrier gas to separate the hydrogen isotopes. This neon, like all waste streams at TSTA, is exhausted into the low pressure holding tank of the TWT to await treatment before being released to the stack. The problem arises in the TWT analytical system, where at the normal GC operating temperature of 100°C, hydrogen and neon are eluted at the same time, and the detector is about 20 times more sensitive to neon. Consequently the computer erroneously calculates a flammable hydrogen concentration whenever neon is present, and the holding tank is unnecessarily swamped with inert gas as a safety measure.

The solution is to program the GC column of the TWT analytical system to run at subambient temperature during elution of neon and the hydrogens. At -40°C there is adequate separation of the hydrogen and neon peaks to avoid the spurious analysis. After elution of the hydrogen the temperature may be rapidly ramped back up to 100°C to quickly elute the high mass components; then the oven can be recooled to be ready for the next sample. Such temperature changes can be easily programmed into a computer-interfaced GC.

CONCLUSIONS

The design philosophies adopted for two state-of-the-art tritium handling systems at Los Alamos have now been tested operationally for significant periods. Tritium operations started at TSTA in June, 1984, and the Salt Facility has now been running for 10 years. Our choices for primary and secondary containment, and for contamination control throughout these installations have proved effective. Despite large inventories at both installations we have had no reportable stack releases and only one instance of a TSF worker receiving an exposure exceeding the quarterly dose allowance.

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